

# PLANT PROTECTION PRODUCT RESIDUES IN APPLES, CAULIFLOWER, CEREALS, GRAPE, LETTUCE, PEAS, PEPPERS, POTATOES AND STRAWBERRIES OF THE SLOVENE ORIGIN IN 2006

## OSTANKI FITOFARMACEVTSKIH SREDSTEV V JABOLKIH, CVETAČI, ŽITIH, GROZDJU, SOLATI, GRAHU, PAPRIKI, KROMPIRJU IN JAGODAH SLOVENSKEGA POREKLA V LETU 2006

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### ABSTRACT

In the year 2006, 181 apple, cauliflower, cereal, grape, lettuce, pea, pepper, potato and strawberry samples from Slovene producers were analysed for plant protection product residues. The samples were analysed for the presence of 86 different active compounds using four analytical methods. In nine samples (5.0 %) exceeded maximum residue levels (MRLs) were determined which is comparable with the results of the monitoring of plant protection product residues in products of plant origin in the European union, Norway, Iceland and Liechtenstein in 2005 (4.9 %).

Key words: GC/MS, LC/MS/MS, plant protection products, plant protection product residues, monitoring

### IZVLEČEK

V letu 2006 smo na ostanke pesticidov analizirali 181 vzorcev jabolk, cvetače, žit, grozdja, solate, graha, paprike, krompirja in jagod slovenskih tržnih pridelovalcev. Vse vzorce smo analizirali s štirimi analitskimi metodami na prisotnost 86 različnih aktivnih spojin. V devetih vzorcih (5,0 %) smo določili presežene maksimalno dovoljene količine ostankov, kar je primerljivo z rezultati monitoringa ostankov pesticidov v rastlinskih proizvodih v Evropski skupnosti, Norveški, Islandiji in Lihtenštajnu v letu 2005 (4,9 %).

Ključne besede: GC/MS, LC/MS/MS, fitofarmacevtska sredstva, ostanki fitofarmacevtskih sredstev, monitoring

## RAZŠIRJENI IZVLEČEK

V letu 2006 smo na Kmetijskem inštitutu Slovenije analizirali 181 vzorcev jabolk, cvetače, žit, grozdja, solate, graha, paprike, krompirja in jagod slovenskih tržnih pridelovalcev. Naključno vzorčenje je potekalo na osmih pridelovalnih območjih: Celje, Koper, Kranj, Nova Gorica, Novo mesto, Murska Sobota, Maribor in Ljubljana. Vzorčenje so opravili kmetijski inšpektorji. Vzorce so odvzeli na polju ali v skladiščih, po poteku karence.

Od 181 analiziranih vzorcev, jih 99 (54,7 %) ni vsebovalo ostankov, oziroma so bili le-ti pod mejo detekcije. 73 vzorcev (40,3 %) je vsebovalo ostanke manjše od maksimalno dovoljenih količin ostankov (MRL). Samo 9 vzorcev (5,0 %) je vsebovalo ostanke nad maksimalnimi dovoljenimi količinami ostankov. Rezultati so predstavljeni na sliki 4.

Analizirali smo 36 vzorcev jabolk: 2 vzorca (5,6 %) sta presegala MRL, 28 vzorcev (77,8 %) je vsebovalo ostanke pod MRL, v 6 vzorcih (16,7 %) ostankov nismo določili.

Analizirali smo 11 vzorcev cvetače: ostankov, ki bi presegali MRL nismo določili, 10 vzorcev (90,9 %) je vsebovalo ostanke pod MRL, v 1 vzorcu (9,1 %) ostankov nismo določili.

Analizirali smo 26 vzorcev žit (pšenica, ječmen, proso, pira, tritikala in koruza): (100,0 %) ostankov nismo določili v nobenem vzorcu.

Analizirali smo 20 vzorcev grozdja: 7 vzorcev (35,0 %) je presegalo MRL, 12 vzorcev (60,0 %) je vsebovalo ostanke nižje od MRL, v 1 vzorcu (5,0 %) ostankov nismo določili.

Analizirali smo 16 vzorcev solate: ostankov, ki bi presegali MRL nismo določili, 1 vzorec (6,3 %) je vseboval ostanke pod MRL, v 15 vzorcih (93,8 %) ostankov nismo določili.

Analizirali smo 4 vzorce graha: ostankov, ki bi presegali MRL nismo določili, 1 vzorec (25,0 %) je vseboval ostanke pod MRL, v 3 vzorcih (75,0 %) ostankov nismo določili.

Analizirali smo 16 vzorcev paprike: ostankov, ki bi presegali MRL nismo določili, 3 vzorci (18,8 %) so vsebovali ostanke pod MRL, v 13 vzorcih (81,3 %) ostankov nismo določili.

Analizirali smo 33 vzorcev krompirja: ostankov, ki bi presegali MRL nismo določili, 2 vzorca (6,1 %) sta vsebovala ostanke pod MRL, v 31 vzorcih (93,9 %) ostankov nismo določili.

Analizirali smo 19 vzorcev jagod: ostankov, ki bi presegali MRL nismo določili, 16 vzorcev (84,2 %) je vsebovalo ostanke pod MRL, v 3 vzorcih (15,8 %) ostankov nismo

določili.

Rezultati so predstavljeni na sliki 1.

V letu 2006 so vzorci jabolk, cvetače, žit, grozdja, solate, graha, paprike, krompirja in jagod vsebovali sledeče aktivne spojine: diklofluanid, difenilamin, krezoksime-metil in zoksamid, vsako v 1 vzorcu (0,6 %), difenokonazol, imidakloprid in miklobutanil vsako v 2 vzorcih (1,1 %), prosimidon in tiakloprid vsako v 3 vzorcih (1,7 %), azoksistrobin, metalaksil in tebufenozid vsako v 4 vzorcih (2,2 %), fenazakvin in fludioksonil vsako v 5 vzorcih (2,8 %), fenheksamid in spirodiklofen vsako v 6 vzorcih (3,3 %), kaptan in klorpirifos vsako v 8 vzorcih (4,4 %), klorotalonil in diazinon vsako v 9 vzorcih (5,0 %), pirimetanil v 12 vzorcih (6,6 %), tolilfluanid v 13 vzorcih (7,2 %), ciprodinil, folpet in fosalon vsako v 19 vzorcih (10,5 %) in ditiokarbamate v 41 vzorcih (22,7 %).

Rezultati so predstavljeni v tabeli 2 in na sliki 3.

Aktivne spojine, ki so presegale MRL so bile: fludioksonil v 1 vzorcu (0,6 %), tolilfluanid v 2 vzorcih (1,1 %) in ciprodinil v 7 vzorcih (3,9 %). Rezultati so predstavljeni v tabeli 3.

## INTRODUCTION

Cereals, fruit and vegetables cannot be produced without the appropriate protection from harmful organisms which, regardless of the needs and wishes of consumers, tend to appear in inappropriate places at inappropriate moments. At present, plant protection is based on the use of plant protection products which, when properly used, assure the most economical way of producing adequate quantities of high quality food. The task of the government is to control if the use of plant protection products is correct, which insures healthy food on the market [1, 2, 10, 11, 19, 26].

Plant protection product residues found in agricultural products produced by Slovene market producers were determined until their placement on the market, i.e. after harvesting, in accordance with the Law on Plant Protection Products and Regulation on Residues of Plant Protection Products Found in and on Agricultural Commodities and Products [24, 25].

Monitoring of plant protection product residues in agricultural products of Slovene market producers until their placement on the market allows determination and control a correct use of plant protection products conforming to the good agricultural practice applied in the conventional, integrated and ecological production and determination of origin and cause of the residues found. Due to the random selection of producers it is possible to evaluate the situation and effectiveness of the

previous measures.

The results are used for:

- determination of harmonisation with the legally prescribed maximal residue level (MRL),
- determination of harmonisation of conventional, integrated and ecological production with good agricultural practice,
- determination of origin or cause of residues determined and
- risk assessment for samples which exceeded MRLs.

Due to the characteristic type of foods consumed by Slovenes (the Slovene Food Basket has not yet been made), plant protection product residues are determined each year in the samples of apples, lettuce and potatoes while the choice of other agricultural products is adjusted to the guidelines indicated in the recommendations issued by European Union [12].

## MATERIALS AND METHODS

Beside apple, lettuce and potato samples, agricultural inspectors took samples of cauliflower, cereals, grapes, peas, pepper and strawberries in 2006. The samples were taken randomly, which means that the farmers growing the relevant plants were chosen by coincidence. The inspectors did not suspect that the agricultural products exceeded MRL. Samples were taken in eight production areas of Slovenia: Celje, Koper, Kranj, Nova Gorica, Novo mesto, Murska Sobota, Maribor and Ljubljana. Agricultural products were taken directly in the field or in storehouses after the expiration of pre-harvest interval of the plant protection products used. When sampling in the field the inspectors avoided the field borders. The units of agricultural products sampled were equally distributed in

the field. For instance, if five lettuce heads were sampled the field was distributed in five areas. From each area one lettuce head was taken. The size of the sample was 2 kg [23].

Legally prescribed MRLs are defined on the basis of field trials in accordance with good agricultural practice [13]. Consideration of the pre-harvest interval and the prescribed way of use of the plant protection products is therefore of key importance.

Samples which came to the laboratory were cut in four quarters containing also external surface. Two opposite quarters were taken for the analysis, with the exception of samples smaller than 25 g, of which entire samples were taken. Prior to the analysis samples were stored frozen at -20°C.

Samples were analysed for the content of selected active substances. For each sample one parallel sample was analysed. In case of exceeded MRL three parallel samples were analysed.

In 2006, residues of 86 different compounds were determined using four different methods:

1. Multiresidual GC/MS method for the determination of 67 compounds: acephate, aldrin, azinphos-methyl, azoxystrobin, bifenthrin, bromopropylate, bupirimate, captan, carbaryl, carbofuran, chlorothalonil, chlorpropham, chlorpyrifos, chlorpyrifos-methyl, cyhalotrin-lambda, cypermethrin, cyprodinil, cyromazine, DDT, deltamethrin, diazinon, dichlofluanid, dimethoate, diphenylamine, endosulfan, endrin, fenitrothion, fenthion, fludioxonil, folpet, HCH- $\alpha$ , heptachlor, heptenophos, imazalil, iprodione, kresoxim-methyl, lindane, malathion, mecarbam, metalaxyl, methamidophos, methidathion, myclobutanil, omethoate, oxydemeton-methyl, parathion, penconazole, permethrin,

Table 1: List of agricultural products of Slovene origin, analysed in 2006, and distribution of sample locations among individual production areas

Tabela 1: Seznam kmetijskih pridelkov slovenskega porekla, analiziranih v letu 2006 in porazdelitev lokacij vzorcev med posameznimi pridelovalnimi območji

Area	Agricultural product									Sum
	Apples	Cauliflower	Cereals	Grapes	Lettuce	Peas	Pepper	Potatoes	Strawberries	
Celje	3	0	2	1	2	2	2	2	2	16
Koper	4	1	1	3	3	0	1	2	0	15
Kranj	1	1	2	0	1	1	0	12	1	19
Ljubljana	2	3	3	0	3	1	2	6	3	23
Maribor	12	1	5	6	2	0	2	6	3	37
Murska Sobota	4	2	10	2	2	0	2	3	2	27
Nova Gorica	1	2	0	6	1	0	2	1	1	14
Novo mesto	9	1	3	2	2	0	5	1	7	30
Sum	36	11	26	20	16	4	16	33	19	181

Table 2: Active substances found in fruit, vegetable and cereal samples of Slovene origin in 2006  
Tabela 2: Najdene aktivne snovi v vzorcih sadja, zelenjave in žit slovenskega porekla v letu 2006

active substance	number of samples										sample portion	
	apples	cauliflower	cereals	grapes	lettuce	peas	pepper	potatoes	strawberries	sum	sum	
azoxystrobin	0	0	0	1	0	0	0	0	3	4	2.2	2.2
captan	8	0	0	0	0	0	0	0	0	8	4.4	4.4
chlorothalonil	0	0	0	7	0	1	1	0	0	9	5.0	5.0
chlorpyrifos	5	0	0	3	0	0	0	0	0	8	4.4	4.4
cyprodinil	1	0	0	9	0	0	0	0	9	19	10.5	10.5
diazinon	9	0	0	0	0	0	0	0	0	9	5.0	5.0
dichlofluanid	0	0	0	0	0	0	0	0	1	1	0.6	0.6
difenoconazole	0	1	0	0	0	0	1	0	0	2	1.1	1.1
diphenylamine	1	0	0	0	0	0	0	0	0	1	0.6	0.6
dithiocarbamates	23	10	0	4	1	0	1	1	1	41	22.7	22.7
fenazaquin	1	0	0	4	0	0	0	0	0	5	2.8	2.8
fenhexamid	0	0	0	5	0	0	0	0	1	6	3.3	3.3
fludioxonil	0	0	0	2	0	0	0	0	3	5	2.8	2.8
folpet	0	0	0	19	0	0	0	0	0	19	10.5	10.5
imidacloprid	0	0	0	0	0	0	2	0	0	2	1.1	1.1
kresoxim-methyl	0	0	0	1	0	0	0	0	0	1	0.6	0.6
metalaxyl	0	0	0	2	0	0	0	0	2	4	2.2	2.2
myclobutanil	1	0	0	1	0	0	0	0	0	2	1.1	1.1
phosalone	14	0	0	2	0	0	0	1	2	19	10.5	10.5
procymidone	0	0	0	0	0	0	0	0	3	3	1.7	1.7
pyrimethanil	6	0	0	3	0	0	0	0	3	12	6.6	6.6
spirodiclofen	5	0	0	1	0	0	0	0	0	6	3.3	3.3
tebufenozide	1	0	0	3	0	0	0	0	0	4	2.2	2.2
thiacloprid	2	0	0	0	0	0	0	0	1	3	1.7	1.7
tolyfluanid	9	0	0	0	0	0	0	0	4	13	7.2	7.2
zoxamide	0	0	0	1	0	0	0	0	0	1	0.6	0.6



Sixteen lettuce samples were analysed: residues exceeding MRLs were not determined, one sample (6.3 %) contained residues lower than MRLs, residues were not found in 15 samples (93.8 %).

Four pea samples were analysed: residues exceeding MRLs were not determined, one sample (25.0 %) contained residues lower than MRLs, residues were not found in 3 samples (75.0 %).

Sixteen pepper samples were analysed: residues exceeding MRLs were not determined, 3 samples (18.8 %) contained residues lower than MRLs, residues were not found in 13 samples (81.3 %).

Thirty-three potato samples were analysed: residues exceeding MRLs were not determined, 2 samples (6.1 %) contained residues lower than MRLs, residues were not found in 31 samples (93.9 %).

Nineteen strawberry samples were analysed: residues exceeding MRLs were not determined, 16 samples (84.2 %) contained residues lower than MRLs, residues were not found in 3 samples (15.8 %).

Results are presented in Figure 1.

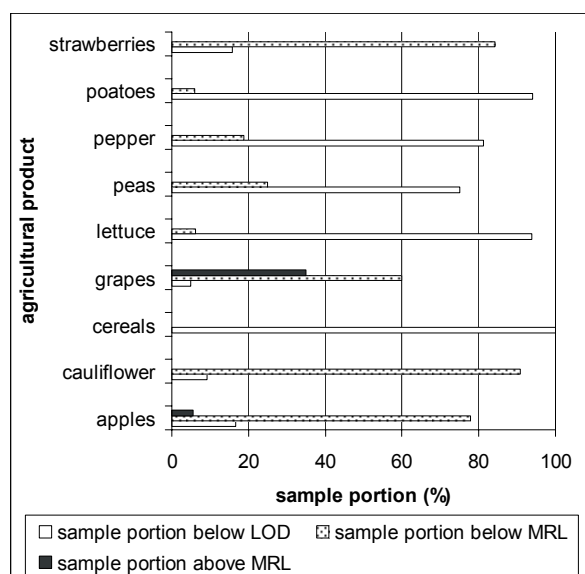


Figure 1: Plant protection product residues in different agricultural products of Slovene origin in 2006

Slika 1: Ostanki fitofarmacevtskih sredstev v različnih kmetijskih pridelkih slovenskega porekla v letu 2006

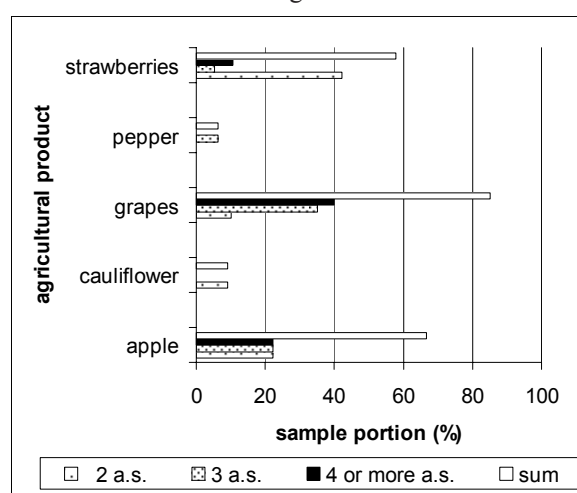
Fifty-four samples (29.8 %) out of 181 samples contained multiple residues. Residues of two active substances were determined in 8 out of 36 apple samples (22.2 %), 1 out of 11 cauliflower samples (9.1 %), 2 out of 20 grape samples (10.0 %) and 8 out of 19 strawberry samples (42.1 %). Residues of three active substances were determined in

8 out of 36 apple samples (22.2 %), 7 out of 20 grape samples (35.0 %), 1 out of 16 pepper samples (6.3 %) and 1 out of 19 strawberry samples (5.3 %). Residues of more than three active substances were determined in 8 out of 36 apple samples (22.2 %), 8 out of 20 grape samples (40.0 %) and 2 out of 19 strawberry samples (10.5 %). In one grape sample nine active substances were found, i.e. the highest number of different residues in one sample.

Contribution of samples with multiple residues was 66.7 % (24 samples) for apples, 9.1 % (1 sample) for cauliflower, 85.0 % (17 samples) for grapes, 6.3 % (1 sample) for pepper and 57.9 % (11 samples) for strawberries.

Residues of one active substance were found in lettuce, pea and potato samples while no residues were found in cereals.

The results are shown in Fig. 2.



a.s. stands for active substances

a.s. pomeni aktivna substanca

Figure 2: Distribution of samples of Slovene origin with multiple plant protection product residues in 2006

Slika 2: Porazdelitev vzorcev slovenskega porekla z dvema ali več ostanki fitofarmacevtskih sredstev v letu 2006

In 2006 apple, cauliflower, cereal, grape, lettuce, pea, pepper, potato and strawberry samples contained the following active substances: dichlofluanid, diphenylamine, kresoxim-methyl and zoxamide each in one sample (0.6 %), difenoconazole, imidacloprid and myclobutanil each in 2 samples (1.1 %), procymidone and thiacloprid each in 3 samples (1.7 %), azoxystrobin, metalaxyl and tebufenozide each in 4 samples (2.2 %), fenazaquin and fludioxonil each in 5 samples (2.8 %), fenhexamid and spirodiclofen each in 6 samples (3.3 %), captan and chlorpyrifos each in 8 samples (4.4 %), chlorothalonil and diazinon each in 9 samples (5.0 %),



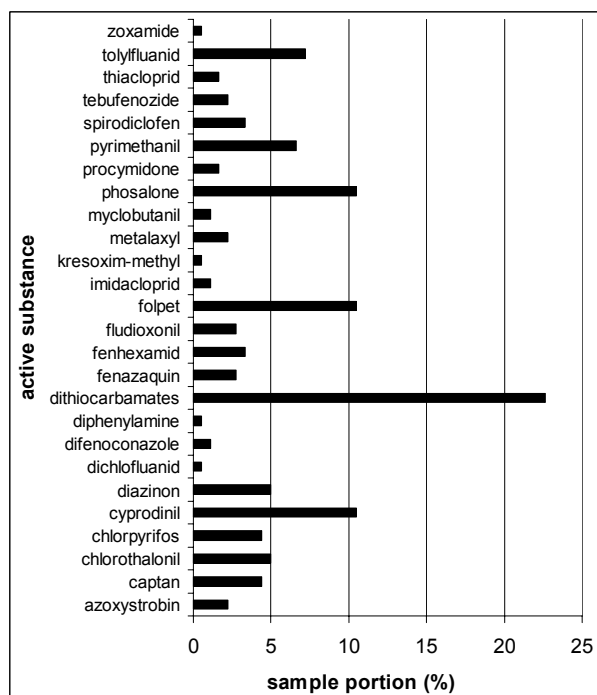


Figure 3: Samples with active substances found in agricultural products of Slovene origin in 2006

Slika 3: Vzorci z aktivnimi spojinami najdenimi v kmetijskih pridelkih slovenskega porekla v letu 2006

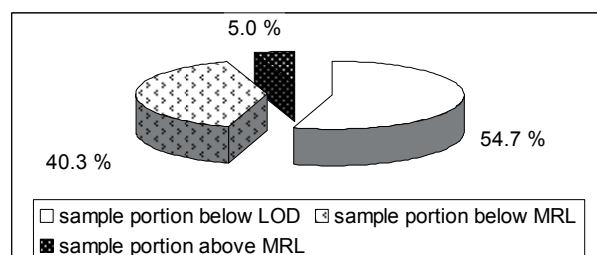


Figure 4: Results of monitoring in Slovenia in 2006

Slika 4: Rezultati monitoringa v Sloveniji v letu 2006

pyrimethanil in 12 samples (6.6 %), tolylfluanid in 13 samples (7.2 %), cyprodinil, folpet and phosalone each in 19 samples (10.5 %) and dithiocarbamates in 41 samples (22.7 %). The residues most frequently found were those from the group of dithiocarbamates: maneb, mancozeb, metiram, propineb and zineb (fungicides) followed by cyprodinil (fungicide), folpet (fungicide) and phosalone (insecticide). The results are given in Table 2 and Fig. 3. Active substances exceeding MRLs were the following: fludioxonil in one sample (0.6 %), tolylfluanid in 2 samples (1.1 %) and cyprodinil in 7 samples (3.9 %). The results are shown in Table 3. The highest cyprodinil content in grape sample was 0.40 mg/kg (MRL is 0.02

mg/kg), fludioxonil content in grape sample was 0.03 mg/kg (MRL is 0.02 mg/kg) and the highest tolylfluanid content in apple sample was 0.26 mg/kg (MRL is 0.21 mg/kg).

The risk assessment performed with Pesticide Safety Directorate (PSD, York, UK), model for acute exposure for cyprodinil at concentration level 0.40 mgkg<sup>-1</sup> and Acceptable Daily Intake (ADI) 0.03 mgkg<sup>-1</sup> body weight<sup>-1</sup> day<sup>-1</sup> (Acute Reference Dose, ARfD, for cyprodinil was not determined) showed that National Estimate of Short Term Intake (NESTI) expressed in ADI percentage ranged from 2.5 % for 7-10 years old children to 31.6 % for adults. The risk assessment performed with PSD model for acute exposure for fludioxonil at concentration level 0.03 mgkg<sup>-1</sup> and ADI 0.40 mgkg<sup>-1</sup> body weight<sup>-1</sup> day<sup>-1</sup> (ARfD for fludioxonil was not determined) showed that NESTI expressed in ADI percentage ranged from 0.0 % for 1-10 years old children and residential elderly people to 0.2 % for adults and vegetarians. The risk assessment performed with PSD model for acute exposure for tolylfluanid at concentration level 0.26 mgkg<sup>-1</sup> and ARfD 0.25 mgkg<sup>-1</sup> body weight<sup>-1</sup> day<sup>-1</sup> showed that NESTI expressed in ARfD percentage ranged from 1.4 % for elderly people living at home and residential elderly people to 10.2 % for infants. ADIs and ARfDs were found on the internet ([http://europa.eu.int/comm/food/plant/protection/evaluation/index\\_en.htm](http://europa.eu.int/comm/food/plant/protection/evaluation/index_en.htm)), [Status of active substances under EU review \(doc. 3010\)](http://www.pesticides.gov.uk/approvals.asp?id=1687) as well as the PSD model for acute exposure (<http://www.pesticides.gov.uk/approvals.asp?id=1687>). The risk assessment showed that the exceeded fruit samples did not present any risk for health (NESTI in % of ADI or ARfD was below 100%) and are therefore safe for the consumers.

Active substances not registered in the Republic of Slovenia were found in apples (diphenylamine), pepper (difenoconazole) and strawberries (dichlofluanid, phosalone, pyrimethanil and thiacloprid) [20].

Active substances not allowed in the integrated production in the Republic of Slovenia were determined in cauliflower (dithiocarbamates), pepper (dithiocarbamates) and strawberries (procymidone) [28-31]. However, attention should be paid to the fact that cauliflower (cruciferous plant) contains naturally present compounds which can produce CS<sub>2</sub> under the conditions of an analysis (dithiocarbamates are determined as CS<sub>2</sub>). Due to the unselective method applied the origin of CS<sub>2</sub> can not be determined and/or it can not be attributed to the use of a plant protection product containing dithiocarbamates.

Active substances not allowed in the ecological production in the Republic of Slovenia were found in apples (dithiocarbamates, myclobutanil) and potatoes (dithiocarbamates).

Table 4: Monitoring results of plant protection product residues in agricultural products of Slovene origin from 2001 to 2006

Tabela 4: Rezultati monitoringa ostankov fitofarmacevtskih sredstev v kmetijskih pridelkih slovenskega porekla od 2001 do 2006

year	sample portion below LOD (%)	sample portion below or equal MRL (%)	sample portion above MRL (%)
2001	72.8	21.9	5.3
2002	46.0	44.0	10.0
2003	56.0	33.3	10.7
2004	53.1	43.1	3.8
2005	68.7	28.7	2.6
2006	54.7	40.3	5.0

Table 5: Monitoring results of plant protection product residues in fruit, vegetables and cereals in European Union, Norway, Iceland and Liechtenstein from 2001 to 2006

Tabela 5: Rezultati monitoringa ostankov fitofarmacevtskih sredstev v sadju, zelenjavi in žitih v Evropski uniji, Norveški, Islandiji in in Lihtenštajnu od 2001 do 2006

year	sample portion below LOD (%)	sample portion below or equal MRL (%)	sample portion above MRL (%)
2001	59	37	3.9
2002	56	38.5	5.5
2003	56	38.5	5.5
2004	53	42	5.0
2005	51	44	4.9

In 2006, 99 samples (54.7 %) out of 181 samples analysed did not contain any residue or their contents were below the limit of detection of the method, 73 samples (40.3 %) contained residues lower or equal to MRLs and 9 samples (5.0 %) contained residues above MRLs (Fig. 4).

The results of monitoring of plant protection product residues in Slovenia obtained from 2001 to 2005 are presented in Table 4 [4, 6, 7]. Results for monitoring of plant protection product residues in European Union, Iceland and Liechtenstein are presented in Table 5 [14-18]. The increased rates of plant protection product residues detections can be partly explained by the enhanced analytical capabilities of the laboratory. This development is reflected by the continuously increasing numbers of active substances sought in the analytical screens. The lowered number of exceedances is probably due to better knowledge of farmers how to properly use plant protection products.

## CONCLUSIONS

Levels of plant protection product residues in agricultural products in Slovenija in 2006 do not give any cause for alarm. 54.7 % samples examined did not contain any residues. Exceeding maximum residue levels were found in 5.0 % samples of agricultural products. The risk assessment for all samples exceeding MRLs showed that

the agricultural products do not represent any risk for health and are therefore safe for the consumers.

For comparison, the results of national monitoring from 2001 to 2005, performed in EU countries and in Norway, Iceland and Liechtenstein, are presented [14-18]. They have shown that 51 % to 59 % of all examined fresh (unprocessed) fruit, vegetable and cereals samples did not contain plant protection product residues, 37 % to 44 % of fresh (unprocessed) samples contained residues lower or equal to MRLs and 3.9 % to 5.5 % of examined fresh (unprocessed) samples contained residues above MRLs. The results for 2006 are not available yet.

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